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METHODS OF TEST FOR PULP

PART XII DETERMINATION OF CALCIUM CONTENT

UDC 676·1 : 676·014·2 : [546·41]

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February 1976

Indian Standard

METHODS OF TEST FOR PULP

PART XII DETERMINATION OF CALCIUM CONTENT

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Indian Standard

METHODS OF TEST FOR PULP

PART XII DETERMINATION OF CALCIUM CONTENT

0. FOREWORD

0.1 This Indian Standard (Part XII) was adopted by the Indian Standards Institution on 30 August 1975, after the draft finalized by the Paper and Its Products (Excluding Packaging Materials) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard is mainly based on ISO/R 777-1968 ' Pulps — Determination of calcium content ', issued by International Organization for Standardization.

0.3 Excessive amounts of calcium salts in pulp have an adverse effect on the quality of paper specially with regard to its sizing characteristics. It is, therefore, essential to know the calcium content in the washed bleached pulp before feeding it to the paper making machine.

0.4 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part XII) prescribes the method for the determination of calcium content of pulp.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960†), freshly boiled and cooled, shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of test.

3. PRINCIPLE OF THE METHOD

3.1 The pulp is ashed and the ash is dissolved in nitric acid. Calcium is determined volumetrically with a solution of the disodium salt of ethylenediaminetetraacetic acid at a *p*H above 12. Other metal ions are masked with triethanolamine.

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*Rules for rounding off numerical values (*revised*).

†Specification for water, distilled quality (*revised*).

4. REAGENTS

4.1 Nitric Acid — 5 M (approx). Dilute 325 ml of nitric acid (r. d. 1·4) to 1 litre with water.

4.2 Hydrochloric Acid — 6 M (approx). Dilute 500 ml of hydrochloric acid (r. d. 1·19) to 1 litre with water.

4.3 Hydrochloric Acid — 1 M. Dilute 82·5 ml of hydrochloric acid (r. d. 1·19) to 1 litre with water.

4.4 Potassium Hydroxide Solution — 8 M (approx). Dissolve 449 g of potassium hydroxide per litre.

4.5 Triethanolamine Solution — Dissolve 100 g of triethanolamine $[(C_2H_5O_3) N]$ in water and dilute to 1 litre.

4.6 Hydroxylamine Hydrochloride Solution — Dissolve 2 g of hydroxylamine hydrochloride ($NH_2OH.HCl$) in 100 ml of water.

4.7 Standard Calcium Solution — 0·01 M. Weigh 1 g of calcium carbonate, dried at a temperature not exceeding 200°C, to the nearest 0·001 g into a 500-ml beaker. Add 100 ml of water and add drop by drop from a burette the smallest (about 20 ml) of 1 M hydrochloric acid, necessary to dissolve the carbonate. Boil out the carbon dioxide, cool, and transfer quantitatively by rinsing with water into a 1 litre volumetric flask and dilute to the mark. Calculate the molarity of the standard calcium solution as follows:

$$M_{\mathrm{Ca}} = \frac{\textit{mass of calcium carbonate in grams}}{100{\cdot}1}$$

4.8 Solution of Disodium Salt of Ethylenediaminetetraacetic Acid (EDTA) — 0·01 M. Dissolve 3·80 g of the disodium salt of ethylenediaminetetraacetic acid ($C_{10}H_{14}O_8N_2Na_2.2H_2O$) in water in a 1-litre volumetric flask and dilute to the mark. Keep the solution in a polyethylene bottle, standardize the solution by adding to a conical flask 20·0 ml of 0·01 M standard calcium solution, 40 ml of water, 4 ml of potassium hydroxide solution, 2 ml of hydroxylamine hydrochloride solution and about 100 mg of cal-red indicator. Titrate also a blank solution containing all the reagents except the standard calcium solution, which is replaced by an equal volume of distilled water.

Calculate the molarity of the EDTA solution from the following equation:

$$M_{\mathrm{EDTA}} = \frac{\mathrm{a} \times M_{\mathrm{Ca}}}{b - c}$$

where

a = volume of the standard calcium solution, expressed in millilitres;

b = volume of the EDTA solution, consumed by the standard calcium solution, expressed in millilitres;

c = volume of the EDTA solution, consumed in the blank determination, expressed in millilitres; and

M_{Ca} = molarity of the standard calcium solution.

The solution mentioned under **4.4**, **4.7** and **4.8** should preferably be stored in polyethylene bottles.

4.9 Cal-Red Indicator — Rub 100 mg of 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthyl-azo) -3-naphthoic acid in a mortar with 10 g of sodium sulphate, to form a homogeneous mixture. Store in a dark bottle.

NOTE — When the sample contains more than 0·03 mg of copper the indicator is blocked. In this case use calcein as the indicator or mask the copper present by addition of approximately 5 ml of 1 percent solution of potassium cyanide.

5. APPARATUS

5.1 Dishes — of quartz, platinum or porcelain.

6. PROCEDURE

6.1 Preparation of Test Sample

6.1.1 Weigh about 10 g of air dry pulp, previously torn to pieces of suitable size, to the nearest 0·01 g.

NOTE — For pulps containing more than 500 mg of calcium per kilogram, weigh an amount of pulp corresponding to a maximum of 5 mg of calcium or use a standardized 0·05 M EDTA solution for the titration.

6.1.2 At the same time weigh out a separate sample for the determination of dry matter content.

6.2 Determination

6.2.1 Ash the test sample in a dish as described in IS : 6213 (Part VII)-1971*.

NOTE — For cleaning of dishes, wash carefully; remove any spots in the dishes by rubbing with fine sand. Boil the dish four times with 6 M hydrochloric acid.

6.2.2 Rinse the inside of the dish with about 10 ml of water and add 3 ml of nitric acid. Heat the dish for 5 to 10 minutes on a steam-bath. If a brown precipitate of manganese dioxide is formed, filter the contents of the dish through a filter paper into a 300-ml conical flask and wash with water. If no insoluble residue is visible or if the residue is colourless, filtration is unnecessary. In that case rinse the contents of the dish directly into a 400-ml conical flask with the aid of water and adjust the volume to between 50 to 100 ml.

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*Methods of test for pulp: Part VII Ash content in pulp.

6.2.3 Add 5 ml of potassium hydroxide solution and add, after 5 minutes with occasional shaking of the flask, 5 ml of triethanolamine solution, 2 ml of hydroxylamine hydrochloride solution and about 100 mg of cal-red indicator. Titrate with the EDTA solution to a colour change from wine-red to pure blue.

NOTE — The sum of calcium and magnesium can be determined at pH 10 by titration with 0·01 M EDTA solution using eriochrome black T as indicator. When more than 0·03 mg of copper is present the indicator is blocked. In this case, add approximately 5 ml of 1 percent solution of potassium cyanide.

6.3 Blank Test — To check that the new reagents do not contain any calcium, titrate a blank solution containing all the reagents and with the sample solution replaced by an equal volume of water.

7. CALCULATION

7.1 Carry out two determinations and calculate the results as follows:

$$\text{Calcium content in milligrams per kilogram (parts per million)} = \frac{(d - e)\, M_{\text{EDTA}} \times 40{\cdot}08 \times 1\,000}{m}$$

where

d = volume in ml of the EDTA solution consumed by the sample,

e = volume in ml of the EDTA solution consumed by the blank solution,

M_{EDTA} = molarity of the EDTA solution, and

m = mass in g of pulp, calculated on an oven-dry basis.

Report the results in milligrams per kilogram (parts per million) as indicated below:

Mean of Duplicate Determinations	*Nearest Unit of Report*
mg/kg	
<100	1
100 to 500	5
>500	10

8. REPORT

8.1 The test report should state the results obtained and moreover, indicate all the conditions of the test, any details of procedure regarded as optional or not laid down in this standard and any incidents that may have affected the results.

INDIAN STANDARDS

ON

METHODS OF TEST FOR PULP, PAPER AND ALLIED PRODUCTS

IS :

1060 (Part I)-1966 Methods of sampling and test for paper and allied products: Part I (*revised*)

1060 (Part II)-1960 Methods of sampling and test for paper and allied products: Part II

1060 (Part III)-1969 Methods of sampling and test for paper and allied products: Part III

2188-1962 Methods of test for paper for electrical purposes

4006 (Part I)-1966 Methods of test for paper and pulp based packaging materials: Part I

4006 (Part II)-1972 Methods of test for paper and pulp based packaging materials: Part II

5285-1969 Methods of test for fibre analysis of paper and board

6213 (Part I)-1971 Methods of test for pulp: Part I Water solubility of pulp

6213 (Part II)-1971 Methods of test for pulp: Part II Determination of freeness of pulp

6213 (Part III)-1971 Methods of test for pulp: Part III Determination of alpha, beta, and gamma cellulose in pulp

6213 (Part IV)-1971 Methods of test for pulp: Part IV Determination of viscosity of pulp

6213 (Part V)-1971 Methods of test for pulp: Part V Solubility of pulp in one percent caustic soda solution

6213 (Part VI)-1971 Methods of test for pulp: Part VI Copper number of pulp

6213 (Part VII)-1971 Methods of test for pulp: Part VII Ash content in pulp

6213 (Part VIII)-1973 Methods of test for pulp: Part VIII Beating, sheet making, preparation of hand sheets and testing

6213 (Part IX)-1973 Methods of test for pulp: Part IX Bleach requirements and preparation of hand sheets for optical tests of pulp

6213 (Part X)-1975 Methods of test for pulp: Part X Determination of the *kappa* number

6213 (Part XI)-1975 Methods of test for pulp: Part XI Determination of acid insoluble ash

6213 (Part XII)-1975 Methods of test for pulp: Part XII Determination of calcium content

6213 (Part XIII)-1975 Methods of test for pulp: Part XIII Determination of copper

6213 (Part XIV)-1975 Methods of test for pulp: Part XIV Determination of iron

6213 (Part XV)-1975 Methods of test for pulp: Part XV Determination of manganese

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